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(12) INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

(19) World Intellectual Property Organization
International Bureau(43) International Publication Date
26 July 2001 (26.07.2001)

PCT

(10) International Publication Number
WO 01/52650 A2(51) International Patent Classification⁷: A01N 25/04

(21) International Application Number: PCT/JP01/00309

(22) International Filing Date: 18 January 2001 (18.01.2001)

(25) Filing Language: English

(26) Publication Language: English

(30) Priority Data:
2000-13459 21 January 2000 (21.01.2000) JP

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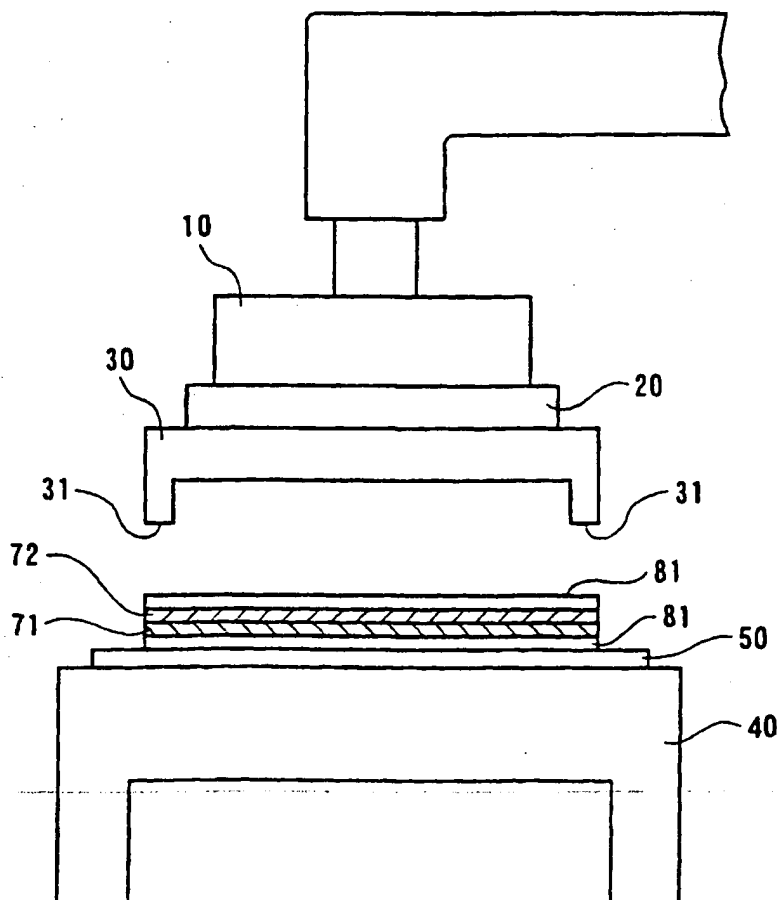
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(81) Designated States (national): AE, AG, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, BZ, CA, CH, CN, CR, CU, CZ, DE, DK, DM, DZ, EE, ES, FI, GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MA, MD, MG, MK, MN, MW, MX, MZ, NO, NZ, PL, PT, RO, RU, SD, SE, SG, SI, SK, SL, TJ, TM, TR, TT, TZ, UA, UG, US, UZ, VN, YU, ZA, ZW.

(84) Designated States (regional): ARIPO patent (GH, GM, KE, LS, MW, MZ, SD, SL, SZ, TZ, UG, ZW), Eurasian

[Continued on next page]

(54) Title: HERBICIDAL MICROEMULSION



(57) Abstract: A microemulsion containing (1) at least one herbicidal active ingredient selected from the group consisting of phenoxyphenoxy carboxylic acids, heteroaryloxyphenoxy carboxylic acids and their derivatives, (2) at least one nonionic surfactant selected from the group consisting of polyoxyalkylene styrylaryl ethers, polyoxyalkylene sorbitan esters, sorbitan alkylates, polyoxyethylene polyoxypropylene alkylphenols and polyoxyalkylene alkyl ethers and (3) water.

WO 01/52650 A2



patent (AM, AZ, BY, KG, KZ, MD, RU, TJ, TM), European
patent (AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE,
IT, LU, MC, NL, PT, SE, TR), OAPI patent (BF, BJ, CF,
CG, CI, CM, GA, GN, GW, ML, MR, NE, SN, TD, TG).

*For two-letter codes and other abbreviations, refer to the "Guid-
ance Notes on Codes and Abbreviations" appearing at the begin-
ning of each regular issue of the PCT Gazette.*

Published:

- without international search report and to be republished
upon receipt of that report

DESCRIPTION

HERBICIDAL MICROEMULSION

BACKGROUND OF THE INVENTIONTECHNICAL FIELD

5 The present invention relates a microemulsion containing at least one herbicidal active ingredient selected from the group consisting of phenoxyphenoxy carboxylic acids, heteroaryloxyphenoxy carboxylic acids and their derivatives.

10 BACKGROUND ART

Formulations of agricultural chemicals have been studied so as to suit the properties of the agricultural active ingredient. For slightly water-soluble agricultural active ingredient, formulations such as
15 emulsifiable concentrate, suspension concentrate, wettable powder, water-dispersible granules are generally used. Particularly, emulsifiable concentrate has been utilized frequently for a long time because it is easy to prepare, easy to handle and is prepared at low cost.

20 However, improvement and replacement of emulsifiable concentrate is demanded increasingly due to various problems attributable to the organic solvent in the formulation, such as toxicity and flammability. For this reason, a microemulsion prepared by replacing most
25 organic solvent in emulsifiable concentrate with water has been proposed to solve the above-mentioned problems. For example, JP-A-1-261312 discloses an aqueous

concentrated microemulsion-based herbicide containing a)
a herbicidal phenoxyphenoxy- or heteroaryloxyphenoxy-
carboxylic acid ester and a herbicidal bentazon salt as
an active ingredient, b) a specific emulsifier or wetting
5 agent, c) a specific organic solvent and d) water.

Further, JP-A-5-201807 discloses a microemulsion
containing a) at least one herbicidal active ingredient
selected from the group consisting of phenoxyphenoxy- and
heteroaryloxyphenoxy- carboxylic acids, b) a specific
10 dispersant, c) a specific emulsifier or a wetting agent,
d) a specific organic solvent and e) water.

However, these microemulsion still have various
problems. Microemulsions are usually transparent or
translucent thermodynamically stable liquid mixtures of
15 mutually incompatible water and oil. Microemulsions are
prepared in order to dissolve or disperse slightly water-
soluble agricultural active ingredients in water-based
solvents with little flammability. Such microemulsions
are supposed to be stable and easy to prepare but usually
20 require much surfactants. Besides, in order to prepare
stable microemulsions, proper choice of surfactant is
necessary. Further, even if a stable microemulsion is
prepared by bringing the respective ingredients into
optimum balance, it could get instable and separate out
25 crystals when diluted with water before application due
to the loss of the optimum balance.

DISCLOSURE OF THE INVENTION

Under these circumstances, as a result of their extensive research to find out a stable microemulsion containing at least one herbicidal active ingredient selected from the group consisting of phenoxyphenoxy carboxylic acids, heteroaryloxyphenoxy carboxylic acids and their derivatives, the present inventors have accomplished the present invention. Namely, the present invention provides a microemulsion containing (1) at least one herbicidal active ingredient selected from the group consisting of phenoxyphenoxy carboxylic acids, heteroaryloxyphenoxy carboxylic acids and their derivatives, (2) at least one nonionic surfactant selected from the group consisting of polyoxyalkylene styrylaryl ethers, polyoxyalkylene sorbitan esters, sorbitan alkylates, polyoxyethylene polyoxypropylene alkylphenols and polyoxyalkylene alkyl ethers and (3) water. The microemulsion is not only a physically stable microemulsion which does not undergo milking or phase separation but also a chemically stable microemulsion with good storage stability of the herbicidal active ingredient, and further shows excellent emulsion stability when diluted with water. The present invention also provides a method of inhibiting the growth of an undesirable plant by using the microemulsion.

BEST MODE FOR CARRYING OUT THE INVENTION

Examples of the phenoxyphenoxy carboxylic acids,

heteroaryloxyphenoxy carboxylic acids and their derivatives (such as salts and various esters) as herbicidal active ingredients in the present invention include 2-[4-(2,4-dichlorophenoxy)phenoxy]propionic acid (common name: diclofop) and its alkyl esters, 2-[4-(4-chlorophenoxy)phenoxy]propionic acid (common name: clofop) and its alkyl esters, 2-[4-(α, α, α -trifluoro-p-tolyloxy)phenoxy]propionic acid (common name: trifop) and its alkyl esters, acetone O-[2-[4-(α, α, α -trifluoro-p-tolyloxy)phenoxy]propionyl]oxime (common name: trifopsime), 4-[4-(α, α, α -trifluoro-p-tolyloxy)phenoxy]pent-2-enoic acid (common name: difenopenten) and its alkyl esters, 2-[4-(4-cyano-2-fluorophenoxy)phenoxy]propionic acid (common name: cyhalofop) and its alkyl esters, 2-[2-(4-(3,5-dichloro-2-pyridyloxy)phenoxy)propionyl]isoxazolidine (common name: isoxapyrifop), 2-[4-(3,5-dichloro-2-pyridyloxy)phenoxy]propionic acid (common name: chlorazifop) and its alkynyl esters, 2-[4-(5-chloro-3-fluoropyridin-2-yloxy)phenoxy]propionic acid (common name: clodinafop) and its alkynyl esters, 2-[4-(5-trifluoromethyl-2-pyridyloxy)phenoxy]propionic acid (common name: fluazifop) and its alkyl esters, 2-[4-(3-chloro-5-trifluoromethyl-2-pyridyloxy)phenoxy]propionic acid (common name: haloxyfop) and its alkyl esters and alkoxyalkyl esters, 2-[4-(6-chloro-1,3-benzoxazol-2-yloxy)phenoxy]propionic acid (common name: fenoxaprop)

and its alkyl esters, 2-[4-(6-chloro-1,3-benzothiazol-2-yloxy)phenoxy]propionic acid (common name: fenthiaprop) and its alkyl esters, 2-isopropylideneaminoxyethyl 2-[4-(6-chloroquinoxalin-2-yloxy)phenoxy]propionic acid (common name: propaquizafop) and 2-[4-(6-chloroquinoxalin-2-yloxy)phenoxy]propionic acid (common name: quizalofop) and its alkyl esters. Some of these compounds have optical isomers, and the present invention covers individual isomers and racemates.

10 Among the phenoxyphenoxy carboxylic acids, heteroaryloxyphenoxy carboxylic acids and their derivatives as the herbicidal active ingredients, particularly preferred are heteroaryloxyphenoxy carboxylic acids and their derivatives, and above all, 15 fluazifop and its alkyl esters are preferable. Further, among them, fluazifop-butyl, in particular fluazifop-P-butyl, or an optically active isomer of fluazifop-butyl, is preferred.

 In the present invention, at least one nonionic 20 surfactant which can form a stable microemulsion, namely at least one nonionic surfactant selected from the group consisting of polyoxyalkylene styrylaryl ethers, polyoxyalkylene sorbitan esters, sorbitan alkylates, polyoxyethylene polyoxypropylene alkylphenols and 25 polyoxyalkylene alkyl ethers, preferably from the group consisting of polyoxyalkylene styrylaryl ethers, polyoxyalkylene sorbitan esters, sorbitan alkylates and

polyoxyethylene polyoxypropylene alkylphenols is used. Their polyoxyalkylene moieties are usually made of a homopolymer or a copolymer or block copolymer (a copolymer or block copolymer is not applied to polyoxyalkylene alkyl ethers) of from 5 to 50 moles, preferably about from 5 to 30 moles, particularly preferably about from 10 to 20 moles of ethylene oxide and/or propylene oxide, and their aryl moieties are usually phenyl or naphthyl. The number of substituents on the styryl group is usually about from 1 to 6, preferably about from 1 to 3. More specific examples of the above-mentioned nonionic surfactants include polyoxyethylene monostyrylphenyl ether, polyoxyethylene distyrylphenyl ether, polyoxyethylene tristyrylphenyl ether, polyoxyethylene polyoxypropylene monostyrylphenyl ether, polyoxyethylene polyoxypropylene distyrylphenyl ether, polyoxyethylene polyoxypropylene tristyrylphenyl ether, polyoxyethylene sorbitan ester, polyoxypropylene sorbitan ester, sorbitan alkylates, polyoxyethylene polyoxypropylene alkylphenols, polyoxyethylene oxoalcohol, polyoxypropylene oxoalcohol, polyoxyethylene stearyl ether and polyoxypropylene stearyl ether.

In the present invention, particularly preferable nonionic surfactants are polyoxyalkylene styrylaryl ethers and polyoxyalkylene sorbitan esters, in particular, polyoxyalkylene styrylaryl ethers. Among them, polyoxyethylene monostyrylphenyl ether, polyoxyethylene

distyrylphenyl ether and polyoxyethylene tristyrylphenyl ether are preferable, and further preferred is polyoxyethylene tristyrylphenyl ether.

In the present invention, one or more other
5 herbicidal active ingredients may be contained to heighten the herbicidal effect. The other herbicidal active ingredients (common names) include 2,4-D, linuron, diuron, metribuzine, cyanazine, bentazon, paraquat, acifluorfen-sodium, fomesafen, lactofen, bromoxynil,
10 flumiclorac-pentyl, fluthiacet-methyl, sulfentrazone, norflurazon, clomazone, clethodim, sethoxydim, tepraloxydim, chlorimuron-ethyl, thifensulfuron-methyl, oxasulfuron, flumetsulam, cloransulam-methyl, imazapyr, imazethapyr, imazaquin, imazamox, glyphosate, glufosinate,
15 trifluralin, pendimethalin, ethalfluralin, alachlor, metolachlor, acetochlor, dimethenamid, flufenacet, fluthiamide and various derivatives (such as salts and esters) thereof. Among them, fomesafen, namely 5-(2-chloro- α, α, α -trifluoro-p-tolyloxy)-N-methylsulfonyl-2-
20 nitrobenzamide, and its salts (such as the sodium salt and potassium salt) are preferred.

In the present invention, various additives such as anionic surfactants, organic solvents, antifoaming agents and other additives may be added optionally.

25 Anionic surfactants are useful to form a stable microemulsion and useful to secure excellent herbicidal effect. Specific examples include fatty acid salts,

alkylsulfosuccinates, polycarboxylates, salts of
alkylsulfuric acid esters, alkyl sulfates, alkylaryl
sulfates, alkyl diglycol ether sulfates, salts of alcohol
sulfuric acid esters, alkyl sulfonates, alkylaryl
5 sulfonates, aryl sulfonates, lignin sulfonates,
alkyldiphenyl ether disulfonates, polystyrene sulfonates,
salts of alkylphosphoric acid esters, alkylaryl
phosphates, styrylaryl phosphates, salts of
polyoxyethylene alkyl ether sulfuric acid esters,
10 polyoxyethylene alkylaryl ether sulfates, salts of
polyoxyethylene alkylaryl ether sulfuric acid esters,
polyoxyethylene alkyl ether phosphates, salts of
polyoxyethylene alkyl ether phosphoric acid esters, salts
of polyoxyethylene alkyl aryl phosphoric acid esters,
15 polyoxyethylene styrylaryl ether sulfates,
polyoxyethylene styrylaryl ether phosphates, salts of
polyoxyethylene styrylaryl ether phosphoric acid esters,
salts of naphthalenesulfonate-formalin condensates, salts
of polyacrylic acid and mixtures of two or more of them.
20 Among these anionic surfactants, preferred are
alkylsulfosuccinates and alkylaryl sulfonates, in
particular dialkylsulfosuccinates and
dodecylbenzenesulfonates.

Organic solvents are useful to form a stable
25 microemulsion. Specific examples include glycols such as
ethylene glycol, propylene glycol, ethylene glycol
monobutyl ether, polyethylene glycol and polypropylene

glycol; alcohols such as octanol, hexanol and cyclohexanol; aromatic solvents (under trade names such as Ipsol (Idemitsu Petrochemical Co., Ltd.), Solvesso (Exxon Chemical) and Exxol (Exxon Chemical)); aliphatic solvents (under a trade name IP solvent (Idemitsu Petrochemical Co., Ltd.)), and among them, glycols and alcohols, in particular ethylene glycol, polyethylene glycol and cyclohexanol are preferable.

With respect to the proportion of the respective ingredients, 100 parts by weight of the microemulsion of the present invention contains from 5 to 50 parts by weight, preferably from 7 to 25 parts by weight, of at least one herbicidal active ingredient selected from the group consisting of phenoxyphenoxy carboxylic acids, heteroaryloxyphenoxy carboxylic acids and their derivatives, from 3 to 40 parts by weight, preferably from 5 to 30 parts by weight, of at least one nonionic surfactant and the balance, usually from 10 to 85 parts by weight, preferably from 15 to 72 parts by weight, of water. When the microemulsion contains other ingredients, it may contain from 5 to 40 parts by weight, preferably from 10 to 30 parts by weight, of other herbicidal active ingredients, from 1 to 40 parts by weight, preferably from 3 to 30 parts by weight, of an anionic surfactant and from 1 to 40 parts by weight, preferably from 3 to 30 parts by weight, of an organic solvent.

The microemulsion of the present invention is

obtainable by mixing the ingredients in an arbitrary order. A stirrer or agitator is usually used for the mixing in its preparation.

The microemulsion of the present invention has
5 excellent herbicidal activity and finds a wide range of applications to crop lands such as upland farms, orchards, mulberry fields and non-crop lands such as forests and farm roads, and can control various noxious weeds, for example, in soybean fields and cotton fields. The
10 microemulsion of the present invention is applied so that the herbicidal active ingredient(s) selected from the group consisting of phenoxyphenoxy carboxylic acids, heteroaryloxyphenoxy carboxylic acids and their derivatives would be applied in an amount of from 10 to
15 500 g, preferably from 50 to 300 g, per hectare, and the other herbicidal active ingredient(s), if contained, would be applied in an amount of from 5 to 2000 g, preferably from 50 to 500 g per hectare. The present invention covers such a method of inhibiting the growth
20 of an undesirable plant by using the microemulsion.

Now, the present invention will be described in further detail with reference to Examples. However, it should be understood that the present invention is by no means restricted to such specific Examples. Firstly,
25 Formulation Examples will be described. In the Formulation Examples "parts" means "parts by weight".

FORMULATION EXAMPLE 1

19.8 parts of fluazifop-P-butyl (purity 93%), 15 parts of polyoxyethylene tristyrylphenyl ether (trade name: Sorpol T-15: Toho Chemical Industry, Co., Ltd.), 10 parts of sodium dialkylsulfosuccinate (trade name: NK-EP-70G, Takemoto Oil&Fats Co., Ltd.) and 55.2 parts of water were mixed by stirring to give a microemulsion.

FORMULATION EXAMPLE 2

The above-mentioned procedure in Formulation Example 1 was followed except that 15 part of NK-EP-70G (trade name) and 50.2 parts of water were used to give a microemulsion.

FORMULATION EXAMPLE 3

19.8 pars of fluazifop-P-butyl (purity 93%), 12 parts of Sorpol T-15 (trade name), 15 parts of NK-EP-70G (trade name), 5 pars of ethylene glycol and 48.2 parts of water were mixed by stirring to give a microemulsion.

FORMULATION EXAMPLES 4 to 15

The above-mentioned procedure in Formulation Example 3 was followed to give microemulsions containing the ingredients shown in Table 1 in the proportions shown in Table 1. In the table, PEG-200 and PEG-400 mean polyethylene glycols manufactured by Nacalai tesque, Inc, IP solvent 1620 means an aliphatic solvent manufactured by Idemitsu Petrochemical Co., Ltd., and Exxol D40 means an aromatic solvent manufactured by Exxon Chemical.

12

Table 1

Ingredient	Proportion					
	4	5	6	7	8	9
Fluazifop-P-butyl	19.8	19.8	19.8	19.8	19.8	19.8
Sorpol T-15	15	15	15	15	15	15
NK-EP-70G	10	11	15	15	15	20
Ethylene glycol	20	25	-	-	-	-
Propylene glycol	-	-	5	10	-	-
PEG-200	-	-	-	-	15	20
PEG-400	-	-	-	-	-	-
Cyclohexanol	-	-	-	-	-	-
IP solvent 1620	-	-	-	-	-	-
Exxol D40	-	-	-	-	-	-
Water	35.2	29.2	45.2	40.2	35.2	25.2

Table 1 (Continued)

Ingredient	Proportion					
	10	11	12	13	14	15
Fluazifop-P-butyl	19.8	19.8	19.8	19.8	19.8	19.8
Sorpol T-15	15	15	15	15	15	15
NK-EP-70G	11	20	15	15	15	15
Ethylene glycol	-	-	-	-	-	-
Propylene glycol	-	-	-	-	-	-
PEG-200	-	-	-	-	-	-
PEG-400	10	20	-	-	-	-
Cyclohexanol	-	-	10	24	-	-
IP solvent 1620	-	-	-	-	10	-
Exxol D40	-	-	-	-	-	10
Water	44.2	25.2	40.2	26.2	40.2	40.2

FORMULATION EXAMPLE 16

19.8 parts of fluazifop-P-butyl (purity 93%), 15 parts of polyoxyethylene tristyrylphenyl ether (trade name: Soprophor BSU, Rhone-Poulenc), 15 parts of NK-EP-70G (trade name), 5 parts of propylene glycol and 45.2 parts of water were mixed by stirring to give a microemulsion.

FORMULATION EXAMPLE 17

2.9 parts of sodium hydroxide was dissolved in 30 parts of water, and 29.4 parts of fomesafen (purity 89%) was added thereto to give an aqueous solution of fomesafen-sodium. Then, the aqueous solution was mixed with 22.7 parts of fluazifop-P-butyl (purity 93%) and 15 parts of Sorpol T-15 (trade name) by stirring to give a microemulsion.

FORMULATION EXAMPLE 18

2.9 parts of sodium hydroxide was dissolved in 25 parts of water, and 29.4 parts of fomesafen (purity 89%) was added thereto to give an aqueous solution of fomesafen-sodium. Then, the aqueous solution was mixed with 22.7 parts of fluazifop-P-butyl (purity 93%), 15 parts of Sorpol T-15 (trade name) and 5 parts of NK-EP-70G (trade name) by stirring to give a microemulsion.

FORMULATION EXAMPLES 19 to 24

The above-mentioned procedure in Formulation Example 18 was followed to give microemulsions containing the ingredients shown in Table 2 in the proportions shown in Table 2.

Table 2

Ingredient	Proportion					
	19	20	21	22	23	24
Fluazifop-P-butyl	22.7	22.7	22.7	22.7	22.7	22.7
Fomesafen	29.4	29.4	29.4	29.4	29.4	29.4
Sodium hydroxide	2.9	2.9	2.9	2.9	2.9	2.9
Sorpol T-15	15	-	-	-	-	-
Soprophor BSU	-	8.5	10	10	10	15
NK-EP-70G	10	15	10	12	20	5
Water	20	21.5	25	23	15	25

FORMULATION EXAMPLE 25

1.45 parts of sodium hydroxide was dissolved in 47.55
5 parts of water, and 14.7 parts of fomesafen (purity 87%)
was added thereto to give an aqueous solution of
fomesafen-sodium. Then, the aqueous solution was mixed
with 11.3 parts of fluazifop-P-butyl (purity 93%), 10
parts of Sorpol T-15 (trade name) and 15 parts of NK-EP-
10 70G (trade name) by stirring to give a microemulsion.

FORMULATION EXAMPLES 26 to 29

The above-mentioned procedure in Formulation Example
25 was followed to give microemulsions containing the
ingredients shown in Table 3 in the proportions shown in
15 Table 3.

Table 3

Ingredient	Proportion			
	26	27	28	29
Fluazifop-P-butyl	11.3	11.3	11.3	11.3
Fomesafen	14.7	14.7	14.7	14.7
Sodium hydroxide	1.45	1.45	1.45	1.45
Sorpol T-15	15	15	-	-
Soprophor BSU	-	-	15	15
NK-EP-70G	5	15	5	10
Water	52.55	42.55	52.55	47.55

FORMULATION EXAMPLE 30

1.45 parts of sodium hydroxide was dissolved in 46.75
5 parts of water, and 14.5 parts of fomesafen (purity 87%)
was added thereto to give an aqueous solution of
fomesafen-sodium. Then, the aqueous solution was mixed
with 11.3 parts of fluazifop-P-butyl (purity 93%), 20
parts of Sorpol T-15 (trade name) and 6 parts of
10 propylene glycol by stirring to give a microemulsion.

FORMULATION EXAMPLES 31 to 35

The above-mentioned procedure in Formulation Example
30 was followed to give microemulsions containing the
ingredients shown in Table 4 in the proportions shown in
15 Table 4.

Table 4

Ingredient	Proportion				
	31	32	33	34	35
Fluazifop-P-butyl	11.3	11.3	11.3	11.3	11.3
Fomesafen	14.5	14.5	14.5	14.5	14.5
Sodium hydroxide	1.45	1.45	1.45	1.45	1.45
Sorpol T-15	-	10	15	-	-
Soprophor BSU	20	-	-	15	15
NK-EP-70G	-	15	5	5	10
Propylene glycol	6	6	6	6	6
Water	46.75	41.75	46.75	46.75	41.75

FORMULATION EXAMPLE 36

1.45 parts of sodium hydroxide was dissolved in 46.75
5 parts of water, and 14.5 parts of fomesafen (purity 87%)
was added thereto to give an aqueous solution of
fomesafen-sodium. Then, the aqueous solution was mixed
with 11.3 parts of fluazifop-P-butyl (purity 93%), 20
parts of a mixture of polyoxyethylene styrylphenyl ether
10 and dodecylbenzensulfonate (trade name: Sorpol 355, Toho
Chemical Industry, Co., Ltd.) and 6 parts of propylene
glycol by stirring to give a microemulsion.

FORMULATION EXAMPLE 37

42.1 parts of water and 11.2 parts of a 10.97% sodium
15 hydroxide aqueous solution were mixed, and 12.5 parts of
fomesafen (purity 89%) was added thereto to give an
aqueous solution of fomesafen-sodium. Then, the aqueous
solution was mixed with 10.1 parts of fluazifop-P-butyl

(purity 93%), 13.4 parts of Soprophor BSU (trade name) and 10.7 parts of NK-EP-70G (trade name) by stirring to give a microemulsion.

FORMULATION EXAMPLE 38

5 15.3 parts of water and 20.1 parts of a 10.97% sodium hydroxide aqueous solution were mixed, and 23.3 parts of fomesafen (purity 89%) was added thereto to give an aqueous solution of fomesafen-sodium. Then, the aqueous solution was mixed with 18.8 parts of fluazifop-P-butyl
10 (purity 93%), 10 parts of Soprophor BSU (trade name) and 12.5 parts of NK-EP-70G (trade name) by stirring to give a microemulsion.

COMPARATIVE FORMULATION EXAMPLE 1

1.3 parts of sodium hydroxide was dissolved in 46.7
15 parts of water, and 14.7 parts of fomesafen (purity 87%) was added thereto to give an aqueous solution of fomesafen-sodium. Then, the aqueous solution was mixed with 11.3 parts of fluazifop-P-butyl (purity 93%), 20
20 parts of polyoxyethylene castor oil (trade name: Alkamuls R/81, Rhone-Poulenc) and 6 parts of propylene glycol by stirring. As a result, instead of a microemulsion, a milky mixture that underwent phase separation after a few hours was obtained.

COMPARATIVE FORMULATION EXAMPLE 2

25 1.3 parts of sodium hydroxide was dissolved in 46.7 parts of water, and 14.7 parts of fomesafen (purity 87%) was added thereto to give an aqueous solution of

fomesafen-sodium. Then, the aqueous solution was mixed with 11.3 parts of fluazifop-P-butyl (purity 93%), 15 parts of Alkamuls R/81 (trade name), 5 parts of NK-EP-70G (trade name) and 6 parts of propylene glycol by stirring. As a result, instead of a microemulsion, a milky mixture that underwent phase separation after a few hours was obtained.

COMPARATIVE FORMULATION EXAMPLE 3

1.3 parts of sodium hydroxide was dissolved in 46.7 parts of water, and 14.7 parts of fomesafen (purity 87%) was added thereto to give an aqueous solution of fomesafen-sodium. Then, the aqueous solution was mixed with 11.3 parts of fluazifop-P-butyl (purity 93%), 20 parts of a polyoxyethylene polyoxypropylene block polymer (trade name: Pepol B-182, Toho Chemical Industry, Co., Ltd.) and 6 parts of propylene glycol by stirring. As a result, instead of a microemulsion, a milky mixture that underwent phase separation immediately after preparation was obtained.

Now, Test Examples will be described.

TEST EXAMPLE 1

The microemulsions prepared as described above in Formulation Examples 1 to 36 were put in 20 ml ampoules, 10 g each, and the ampoules were sealed and stored at 20°C for a predetermined period (1 day, 1 week, 2 weeks or 1 month), and the contents were observed from the outside with the naked eye. All the microemulsions

remained stable without phase separation or milking.

TEST EXAMPLE 2

The microemulsions prepared as described above in Formulation Examples 4, 5, 9, 11, 13, 18 to 22, 24 and 26 to 36 were put in 20 ml ampoules, 10 g each, and the ampoules were sealed and stored at -5°C for a predetermined period (1 week, 2 weeks or 1 month), and the contents were observed from the outside with the naked eye. All the microemulsions remained stable without phase separation or milking.

TEST EXAMPLE 3

The microemulsions prepared as described above in Formulation Examples 4, 5 and 11 were put in 20 ml ampoules, 10 g each, and the ampoules were sealed and stored at 5°C for 2 weeks, and the contents were observed from the outside with the naked eye. All the microemulsions remained stable without phase separation or milking.

TEST EXAMPLE 4

The microemulsions prepared as described above in Formulation Examples 1, 2, 4, 5, 8, 9, 11, 13 and 18 to 35 were put in 20 ml ampoules, 10 g each, and the ampoules were sealed and stored at 50°C for 1 day, at 54°C for 2 weeks or at 60°C for 1 week, and the contents were observed from the outside with the naked eye. All the microemulsions remained stable without phase separation or milking.

TEST EXAMPLE 5

The microemulsions prepared as described above in Formulation Examples 13, 20 to 24, 26, 28 and 29 were put in 20 ml ampoules, 10 g each, and the ampoules were
5 sealed and subjected to a cycle test (a total of 21 days of storage comprising three cycles of 3 days at -5°C, 1 day at room temperature and 3 days at 50°C), and the contents were observed from the outside with the naked eye. All the microemulsions remained stable without
10 phase separation or milking.

TEST EXAMPLE 6

Crabgrass (Digitaria sanguinalis L.) was sown in 1/1,000,000 ha pots filled with upland field soil, and when the crabgrass grew to a 2.7 to 3 leaf stage, the
15 microemulsions prepared as described above in Formulation Examples 5 and 11 were so weighed out as to contain predetermined amounts of the active ingredient and diluted with water in an amount of 200 l/ha, and foliage treatment was done. The aqueous diluted solutions were
20 in well-dispersed states.

3 Weeks after the treatment, the crabgrass was checked for growth with the naked eye, and the herbicidal effects were evaluated on a scale of growth inhibition rates (%) from 0 (equivalent to control) to 100 (complete
25 death). The results were shown in Table 5.

Table 5

Formulation Example	Dose of Fluazifop-P-butyl (g/ha)	Growth inhibition rate (%)
5	100	89
	50	87
	25	80
11	100	89
	50	88
	25	75

TEST EXAMPLE 7

Soybean (Glycine max Merr.) was sown in 1/300,000 ha
 5 pots filled with upland field soil, and when the soybean
 grew to a 0.5 to 0.7 leaf stage, the microemulsions
 prepared as described above in Formulation Examples 5 and
 11 were so weighed out as to contain predetermined
 amounts of the active ingredient (200 g or 400g/ha of
 10 fluazifop-P-butyl) and diluted with water in an amount of
 200 l/ha. 0.2 vol% of an agricultural spreader was added
 to the aqueous diluted solutions, and foliage treatment
 was done. The aqueous diluted solutions were in well-
 dispersed states. Two weeks after the treatment, the
 15 soybean was checked for growth with the naked eye, and no
 injury was observed.

TEST EXAMPLE 8

Crabgrass (Digitaria sanguinalis L.), common ragweed
 (Ambrosia artemisiifolia L.) and soybean (Glycine max
 20 Merr.) were sown in an upland field (1 test block: 5 m ×
 1 m), and other weeds were allowed to grow spontaneously.

When the plants grew to predetermined leaf stages (① a 0.5 to 3.5 leaf stage for crabgrass, ② a 0.2 to 2.0 leaf stage for common ragweed, ③ a 0.5 to 3.1 leaf stage for redroot pigweed (Amaranthus retroflexus L.) and ④ a 0.4 leaf stage for soybean), the microemulsions prepared as described above in Formulation Examples 37 and 38 were so weighed out as to contain predetermined amounts of the active ingredient and diluted with water in an amount of 250 l/ha, and foliage treatment was done. The aqueous diluted solutions were in well-dispersed states.

29 days after the treatment, each plant was checked for growth with the naked eye, and the herbicidal effects were evaluated on a scale of growth inhibition rates (%) from 0 (equivalent to control) to 100 (complete death). The results were shown in Table 6.

Table 6

Formulation Example	Dose (g/ha)	Growth inhibition rate (%)			
	Fluazifop-P-butyl+fomesafen	Crabgrass	Common ragweed	Redroot pigweed	Soybean
37	100+125	83	78	95	0
	150+188	85	80	98	8
	200+250	88	93	100	5
38	100+125	80	68	93	0
	150+188	90	80	100	10
	200+250	90	78	100	13

INDUSTRIAL APPLICABILITY

The microemulsion of the present invention is not

only a physically stable microemulsion which does not undergo milking or phase separation but also a chemically stable microemulsion with good storage stability of the herbicidal active ingredient, and further shows excellent
5 emulsion stability when diluted with water. The present invention also provides a method of inhibiting the growth of an undesirable plant by using the microemulsion.

CLAIMS

1. A microemulsion containing (1) at least one herbicidal active ingredient selected from the group consisting of phenoxyphenoxy carboxylic acids,
5 heteroaryloxyphenoxy carboxylic acids and their derivatives, (2) at least one nonionic surfactant selected from the group consisting of polyoxyalkylene styrylaryl ethers, polyoxyalkylene sorbitan esters, sorbitan alkylates, polyoxyethylene polyoxypropylene
10 alkylphenols and polyoxyalkylene alkyl ethers and (3) water.
2. The microemulsion according to Claim 1, which contains at least one nonionic surfactant selected from the group consisting of polyoxyalkylene styrylaryl ethers,
15 polyoxyalkylene sorbitan esters, sorbitan alkylates and polyoxyethylene polyoxypropylene alkylphenols.
3. The microemulsion according to Claim 1, which further contains another herbicidal active ingredient.
4. The microemulsion according to Claim 1, which
20 contains 5-(2-chloro- α, α, α -trifluoro-p-tolyloxy)-N-methylsulfonyl-2-nitrobenzamide or a salt thereof as another herbicidal active ingredient.
5. The microemulsion according to Claim 1 or 3, which further contains an anionic surfactant.
- 25 6. The microemulsion according to Claim 1 or 3, which further contains an organic solvent.
7. The microemulsion according to Claim 1 or 3, which

further contains an anionic surfactant and an organic solvent.

8. A microemulsion containing (1) from 5 to 50 parts by weight of at least one herbicidal active ingredient
5 selected from the group consisting of phenoxyphenoxy carboxylic acids, heteroaryloxyphenoxy carboxylic acids and their derivatives, (2) from 3 to 40 parts by weight of at least one nonionic surfactant selected from the group consisting of polyoxyalkylene styrylaryl ethers,
10 polyoxyalkylene sorbitan esters, sorbitan alkylates, polyoxyethylene polyoxypropylene alkylphenols and polyoxyalkylene alkyl ethers and (3) at least water as the balance.
9. A method of inhibiting the growth of an undesirable
15 plant by using the microemulsion as defined in Claim 1, 3 or 8.

CORRECTED VERSION

(19) World Intellectual Property Organization
International Bureau



(43) International Publication Date
26 July 2001 (26.07.2001)

PCT

(10) International Publication Number
WO 01/52650 A2

(51) International Patent Classification⁷: A01N 25/04

(21) International Application Number: PCT/JP01/00309

(22) International Filing Date: 18 January 2001 (18.01.2001)

(25) Filing Language: English

(26) Publication Language: English

(30) Priority Data:
2000-13459 21 January 2000 (21.01.2000) JP

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(81) Designated States (national): AE, AG, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, BZ, CA, CH, CN, CR, CU, CZ, DE, DK, DM, DZ, EE, ES, FI, GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MA, MD, MG, MK, MN, MW, MX, MZ, NO, NZ, PL, PT, RO, RU, SD, SE, SG, SI, SK, SL, TJ, TM, TR, TT, TZ, UA, UG, US, UZ, VN, YU, ZA, ZW.

(84) Designated States (regional): ARIPO patent (GH, GM, KE, LS, MW, MZ, SD, SL, SZ, TZ, UG, ZW), Eurasian patent (AM, AZ, BY, KG, KZ, MD, RU, TJ, TM), European patent (AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE, TR), OAPI patent (BF, BJ, CF, CG, CI, CM, GA, GN, GW, ML, MR, NE, SN, TD, TG).

Published:

— without international search report and to be republished upon receipt of that report

(48) Date of publication of this corrected version:
27 September 2001

(15) Information about Correction:
sec PCT Gazette No. 39/2001 of 27 September 2001, Section II

For two-letter codes and other abbreviations, refer to the "Guidance Notes on Codes and Abbreviations" appearing at the beginning of each regular issue of the PCT Gazette.



WO 01/52650 A2

(54) Title: HERBICIDAL MICROEMULSION

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(19) World Intellectual Property Organization
International Bureau



(43) International Publication Date
26 July 2001 (26.07.2001)

PCT

(10) International Publication Number
WO 01/52650 A3

(51) International Patent Classification: **A01N 25/04**

(21) International Application Number: **PCT/JP01/00309**

(22) International Filing Date: 18 January 2001 (18.01.2001)

(25) Filing Language: English

(26) Publication Language: English

(30) Priority Data:
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(84) Designated States (regional): ARIPO patent (GH, GM, KE, LS, MW, MZ, SD, SI, SZ, TZ, UG, ZW), Eurasian patent (AM, AZ, BY, KG, KZ, MD, RU, TJ, TM), European patent (AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE, TR), OAPI patent (BF, BJ, CF, CG, CI, CM, GA, GN, GW, ML, MR, NE, SN, TD, TG).

Published:

with international search report

(88) Date of publication of the international search report:
7 February 2002

(15) Information about Correction:

Previous Correction:

see PCT Gazette No. 39/2001 of 27 September 2001, Section II

For two-letter codes and other abbreviations, refer to the "Guidance Notes on Codes and Abbreviations" appearing at the beginning of each regular issue of the PCT Gazette.



WO 01/52650 A3

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INTERNATIONAL SEARCH REPORT

International Application No

PCT/JP 01/00309

A. CLASSIFICATION OF SUBJECT MATTER
IPC 7 A01N25/04

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

IPC 7 A01N

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

WPI Data, EPO-Internal, CHEM ABS Data

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Patent family members are listed in annex.

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Date of the actual completion of the international search

20 September 2001

Date of mailing of the international search report

02/10/2001

Name and mailing address of the ISA

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Decorte, D

INTERNATIONAL SEARCH REPORT

International Application No
PCT/JP 01/00309

C.(Continuation) DOCUMENTS CONSIDERED TO BE RELEVANT

Category	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
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